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Quarterly Progress Report

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**PREPARATION AND EVALUATION
OF HIGH PURITY BERYLLIUM**

by

G. F. Spangler
M. Herman
E. J. Arndt
D. B. Hoover
V. V. Damiano

October 2, 1962 to January 1, 1963

Prepared for

**DEPARTMENT OF THE NAVY
Bureau of Naval Weapons**

Contract No. N0w 62-0536-d

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1. INTRODUCTION

The purpose of this investigation is to produce high purity beryllium and to study its deformation and fracture characteristics.

2. ZONE REFINING

During the past quarter, the zone melting of one one inch diameter SR Pechiney (vacuum cast and extruded) bar was completed and the zone melting of another similar bar begun. The bar that was completed was the second one inch bar of this particular grade starting material to be zone melted in our laboratories. A total of eleven passes were made through this bar at a rate of one half inch per hour. The bar just started received four passes, also at a rate of one half inch per hour, during this period.

3. SINGLE CRYSTAL STUDIES

3.1 Cu-Be Alloy

A single crystal specimen of a copper-beryllium alloy containing 5 weight per cent copper was tested in tension with the crystal oriented for basal glide. This specimen exhibited a CRSS on the basal plane of 7800 PSI and underwent a 19% reduction in area, equivalent to 23% elongation, over about one half the gage length of the specimen prior to fracture. The resolved shear stress on the basal plane at fracture was 12,200 PSI. The other half of the gage length underwent no deformation (no visible slip lines), indicating that the CRSS in this region was at least 9200 PSI. This rather wide difference in yield stress can presumably be attributed to non-uniform distribution of copper along the length of the bar.

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There was no evidence of any prism glide, with the resolved shear stress reaching, at the point of fracture, 7400 PSI on the prism plane most favorably oriented for slip. The fracture, however, did occur by a combination of both basal plane and $(11\bar{2}0)$ prism plane cleavage, forming a "W" shaped interface consisting of two basal plane and two prism plane facets.

This particular alloy was produced by first wrapping 10 mil, high purity copper wire on a seven pass one quarter inch SR Pechiney bar and then melting the copper into the bar by passing a molten zone along the length of the bar. The distance between turns of the copper wire was calculated to produce the desired 5 w/o composition. In this case, a distance of roughly one eighth of an inch between turns was used. A second zone pass was made after the copper was melted in, in an attempt to zone level any regions of non-uniform alloy concentration along the length of the bar.

3.2 Evaluation of Stress for Twinning

A tensile test was also performed on a nine pass SR Pechiney single crystal oriented with the c-axis parallel to the tensile axis. This test was performed to evaluate the effect of purification on the stress for twinning. The specimen fractured by basal cleavage at an applied stress of 8650 PSI with no evidence of yielding or twinning on the load-deformation plot. Post fracture microscopic examinations of the specimen, however, showed that considerable twinning had occurred in the region of the fracture. A majority of this twinning could be attributed to post fracture deformation from the observation that many of the twins appeared to emanate from various small secondary cleavage cracks adjacent to the primary cleavage interface. At least one fairly wide twin appeared to have occurred prior to fracture, however, based on the observation that the major cleavage interface cut through a wide portion of the twin and that the matching halves of the twin on either side of the cleavage were of same width. If this observation is a true

representation of the facts, these results indicate that purification has reduced the stress for twinning to roughly one half the value observed by Tuer and Kaufmann⁽¹⁾ under identical test conditions on relatively impure beryllium.

A second specimen, of the same orientation, was tested in the hope of getting more positive evidence of this reduction in the stress for twinning. The specimen, however, fractured at a shoulder by basal cleavage with no evidence of twinning. Because of this very critical tendency of specimens of this orientation to cleave so readily, without clear cut evidence of twinning prior to fracture, it was decided not to make any further tests of this sort.

4. POLYCRYSTALLINE STUDIES

4.1 Evaluation of Cu-Be Alloy

During the past quarter, a sample of the 5 w/o Cu-Be crystal was hot swaged at 525°C. The as-swaged wire was unrecrystallized and showed a definite $\langle 10\bar{1}0 \rangle$ wire texture. The recrystallization and grain growth characteristics of this wire were determined and found to be only slightly slower than observed in the previous 12PB (twelve pass Brush) hot swaged wire.

Bend tests of 5 w/o Cu-Be wire specimens annealed at 750°C and 900°C, showed the same behavior exhibited by the unalloyed hot swaged beryllium. The specimen annealed at 750°C was brittle; the specimen annealed at 900°C underwent a small amount of bending before fracture. The amount of deformation in both cases was comparable to the unalloyed beryllium.

Tensile tests were performed on a sample of the 12PB, hot-swaged unalloyed beryllium, and on a sample of the 5 w/o Cu-Be alloy. Both were annealed at 750°C to produce mean grain sizes of approximately 50 microns. The 12PB sample yielded at 32,000 PSI and exhibited about

(1) G. L. Tuer and A. R. Kaufmann, "The Metal Beryllium", 1955, Cleveland, Ohio (ASM).

1% extension prior to failure. The 5 w/o Cu-Be specimen yielded at 35,480 PSI and also exhibited about 1% extension, fracturing, however, at a shoulder. The yield stress observed in the 5 w/o Cu-Be specimen was disappointingly low, considering the measured CRSS of 7800 PSI for basal glide observed in this alloy. It is possible, however, that this low value is a result of non-uniform copper distribution, as demonstrated in the yielding behavior of the 5 w/o Cu-Be single crystal tensile specimen.

4.2 Metallographic Studies

Metallographic studies of the inter-crystalline fracture phenomenon observed in the hot swaged wire were continued. One interesting facet of this recent work was the observation of what appeared to be micro-cracks in polished and etched samples of the deformed polycrystalline wire tensile specimens. These micro-cracks, which appeared only upon etching, occurred both at grain boundaries as well as within the grains. The fact that these micro-cracks were observed only in deformed specimens and after etching suggested that the micro-cracks might merely be an etching phenomenon at regions of very localized strain. One half of a broken tensile specimen, the other half of which exhibited the micro-cracks, was then annealed at 900°C for 30 minutes. This latter specimen, upon polishing and etching, exhibited no micro-cracks. It was concluded from this that the micro-cracks previously observed were in reality an etching phenomenon at regions of localized strains.

4.3 Swaging of Zone Refined SR Pechiney Beryllium

- A single crystal sample of our first zone refined one inch diameter (vacuum cast and extruded) SR Pechiney beryllium bar* was hot swaged at 475°C. This particular sample, after swaging, was found to have a very irregularly shaped cross section. It appeared as if the sample initially tended to flatten out and then was curled inward on subsequent deformation. The as-swaged microstructure was found to be

* Hereafter designated 1" SR-1.

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quite coarse-grained and rather moderately twinned. This structure, when annealed, did not appear to recrystallize; the twins merely disappeared with the grain size remaining the same or exhibiting growth, depending upon the annealing temperature.

Whether this rather marked difference in swaging characteristics from the twelve pass Brush samples is a result of the higher purity level of the 1" ~~SA~~-1 bar (basal CRSS of 316 PSI vs. 400 PSI) or because of the slightly higher swaging temperature (475°C vs. 425°C) is not known. Additional samples will be swaged at lower temperatures.

This particular sample was sectioned from the one inch diameter bar by first cutting out a segment 1-1/8" long from the bar. This cylindrical segment was then sectioned longitudinally into seven smaller specimens 1-1/8" long and roughly 1/4" on a side. These sections were subsequently spark machined to swaging samples 1/4" in diameter and 1-1/8" long.

5. ELECTRON TRANSMISSION MICROSCOPY

Polycrystalline beryllium specimens which were prepared by hot rolling zone refined single crystals were studied using electron transmission microscopy techniques. Special attention was paid to the arrangement and distribution of dislocations in the grain boundaries and sub-boundaries, and within the grains and sub-grains. The specimens studied included 12PB-A, 12PB-B, 12PB-C and 12PB-D. Specimens in the 12PB-A group were deformed in tension and compression in the bulk form and then examined in the electron microscope. Other samples were deformed in the electron microscope. One sample prepared by powder techniques was also examined. The quantity and distribution of precipitates or inclusions was also noted in all specimens examined.

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(a) Specimens 12PB-A and 12PB-B (reduced 90% and 50% respectively at 600°C) appeared to have very similar structures. Numerous grain boundaries were visible, and individual dislocations could not be resolved within them. The grains were subdivided into sub-grains, the order of two to five microns in size. The density of dislocations within the sub-grains was very low.

Specimens deformed in the electron microscope exhibited a breaking up of the sub-boundaries as dislocations glided away from the boundaries.

Specimens cut from compressed bulk crystals exhibited large numbers of dislocations within the grains. Some of the grains exhibited dislocation loops which were similar in aspect to loops observed for prism slip.

Distribution of precipitates was non-uniform. Some precipitates were distributed along grain boundaries. Others were precipitated randomly throughout grains.

(b) Specimen 12PB-C (reduced 50% at 725°C) and specimen 12PB-D (reduced 50% at 850°C) both exhibited low angle boundaries in which individual dislocations within the boundaries were easily resolved. These consisted of hexagonal nets lying on the (0001) plane and tilt boundaries. X-ray Laue photographs of 12PB-C taken parallel and perpendicular to the rolling plane suggested that the sub-grains were misoriented by twists about the c-axis. These results were consistent with the observations made in the electron microscope of twist and tilt boundaries. The grain size of both 12PB-C and 12PB-D was appreciably larger than 12PB-A or 12PB-B. No grain boundaries were observed in the field of view observed.

(c) Specimens deformed to failure in the electron microscope exhibited both intergranular and transgranular failures. Failures did not necessarily occur at precipitation sites.

(d) Examination of the specimen prepared from a hot pressed powder compact exhibited numerous grain boundaries. No sub-boundaries were observed within the grains. Individual dislocations were visible within the grains. Their density varied from grain to grain. Large precipitates or inclusions were observed along the grain boundaries and also within the grains.

6. SELF-DIFFUSION IN BERYLLIUM

6.1 Compound Diffusant Source and Diffusion Concentration Profile

6.1.1. General Consideration of the Solutions of the Diffusion Equation

The process of self-diffusion in solids may be described by the simplest form of Fick's second law. For one dimensional diffusion it can be written as:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial X^2} \quad (1)$$

where:

C = the concentration of diffusing atoms,

t = time for diffusion,

D = the diffusivity,

X = distance along the diffusion direction.

The solution of Equation 1 for the boundary condition of a constant surface concentration diffusing into a semi-infinite medium is:

$$C = C_0 \operatorname{erfc} \left(\frac{X}{2 \sqrt{Dt}} \right) \quad (2)$$

where:

C₀ is the surface concentration and

$$\operatorname{erfc}\left(\frac{x}{2\sqrt{Dt}}\right) = 1 - \frac{1}{\sqrt{\pi Dt}} \int_0^{\frac{x}{2\sqrt{Dt}}} \exp\left(-\frac{x^2}{4Dt}\right) dx.$$

For the condition that the total amount of diffusant remains constant but is initially at the surface ($X = 0$), the solution of equation (1) for a semi-infinite geometry is:

$$C = Q/\sqrt{\pi Dt} \exp(-X^2/4Dt). \quad (3)$$

Where

Q is the total number of diffusing atoms.

Equation (2) has been frequently used for diffusion studies in dilute alloys and in systems where the diffusants are from vapor sources; here, the constant surface concentrations may be established experimentally. Since radioactive isotopes have been available for diffusion studies, a thin tracer source may be electroplated onto the surface of the sample for diffusion. Should the loss of the tracers by evaporation be prevented, the boundary condition for Equation (3) will be satisfied experimentally. Therefore, Equation (3) is most frequently applied to metallic diffusion studies along with the tracer techniques.

In order to use radioactive beryllium (Be^7) in an efficient way and avoid the complexities of beryllium electroplating, aqueous $\text{Be}^7 \text{Cl}_2$ solution is deposited onto the surface of the sample and evaporated to dryness to act as a tracer source in the present investigation. Dissociation of $\text{Be}^7 \text{Cl}_2$ or interchange between Be^7 atoms and Be^9 atoms from the sample is expected to occur at the surface of the sample during diffusion at high temperatures. Consequently Be^7 diffusion into the bulk of Be^9 can proceed. Since the rate of reaction depends on the experimental conditions, the boundary condition of this diffusion process varies with experimental conditions (temperatures and times). For example, at low

temperatures, the dissociation rate should be small. If the diffusion time is kept short, a constant surface concentration for diffusion may be established. The concentration distribution will then obey Equation (2). At high temperatures, however, the reaction may be completed within a very short time. Consequently, all the Be^7 atoms are available for diffusion immediately and the boundary condition of Equation (3) will be fulfilled. Therefore, a Gaussian distribution, Equation (3) can be used to determine the diffusion coefficient. Above all, the boundary conditions which will experimentally give the latter distribution can be obtained by a process of pre-annealing and subsequent polishing off of the reaction zone. This type of preparation of the diffusion samples would be applicable to all diffusion experiments of this sort.

6.1.2. Characteristics of Beryllium Radioactive Isotopes

Beryllium has two artificial radioactive isotopes. They are Be^7 and Be^{10} . Be^7 has been produced by cyclotron activation of Li in the form of chloride. Be^7 has 0.48mev emission with a half life time of about 53 days. As it is produced, the specific radioactivity is carrier free. Since the half life time and energy of emission are well within a measurable range, Be^7 is therefore a suitable tracer for diffusion measurements. Be^{10} emits 0.557 mev particles with a half life time of 2.5×10^6 years. At the present time, the Be^{10} commercially available has a low specific activity of about 10^7 dPM/gr. In view of the characteristics of Be^{10} emission, it should also be an excellent self-diffusion tracer providing that some improvement can be made in the specific activity.

6.1.3. Experimental Procedures and Results

Specimens were prepared from a zone refined beryllium single crystal. A piece of the single crystal was first sliced along the basal plane by means of spark discharge machining and then cut into rectangular slabs with dimensions approximately 6 x 5 x 3 mm. The slabs were ground to a final thickness of about 2.5 mm and subjected to a light chemical etching. The flatness of the finishing faces of the specimens was

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measured within 2 microns on a Starrett dial gauge. The two faces of the finished specimen were within three degrees of the basal plane.

Radioactive beryllium chloride was deposited on the surface of the specimen with an initial concentration of no more than one micro curie - or less than 4×10^{11} fresh $\text{Be}^7 \text{Cl}_2$ molecules. The chemical concentration of Be^7 on the specimen is therefore approximately two thousandth of one atomic layer. A couple of the specimens were placed face to face in a tantalum lined stainless steel specimen holder. The holder was then sealed in a quartz capsule under an argon atmosphere. Diffusion anneals were carried out at both 804°C and 607°C with the corresponding diffusion time of 4 and 16 hours.

After the diffusion anneal, approximately 0.5 mm was ground from the back and the four side faces of the specimen. Sectioning was then carried out by means of wet grinding, taking special care to maintain surface flatness. The radioactivity of each section was measured on a 4π geometry scintillation counter by counting the grinding paper. It was also measured on a 2π geometry scintillation counter by counting the residual activity of the specimen after each sectioning operation. The thickness of each section was determined from the measurement of the specimen thickness after each new grinding.

The concentration profiles for the diffusion experiments at 804°C and 607°C are shown in Figures 1 and 2 respectively. On each figure, two curves are presented to represent the two methods of measurement of radioactivity as mentioned previously. The plots are self-consistent. It can be concluded that the loss of beryllium during sectioning is negligible.

Diffusivities are calculated from Equation (3) by re-plotting the concentration profiles (Figs. 1 and 2) as $\log C$ vs. X^2 in Figures 3 and 4. Because Figure 3 shows a nearly straight line relation over its entire length, the concentration profile shown in Figure 1 should be

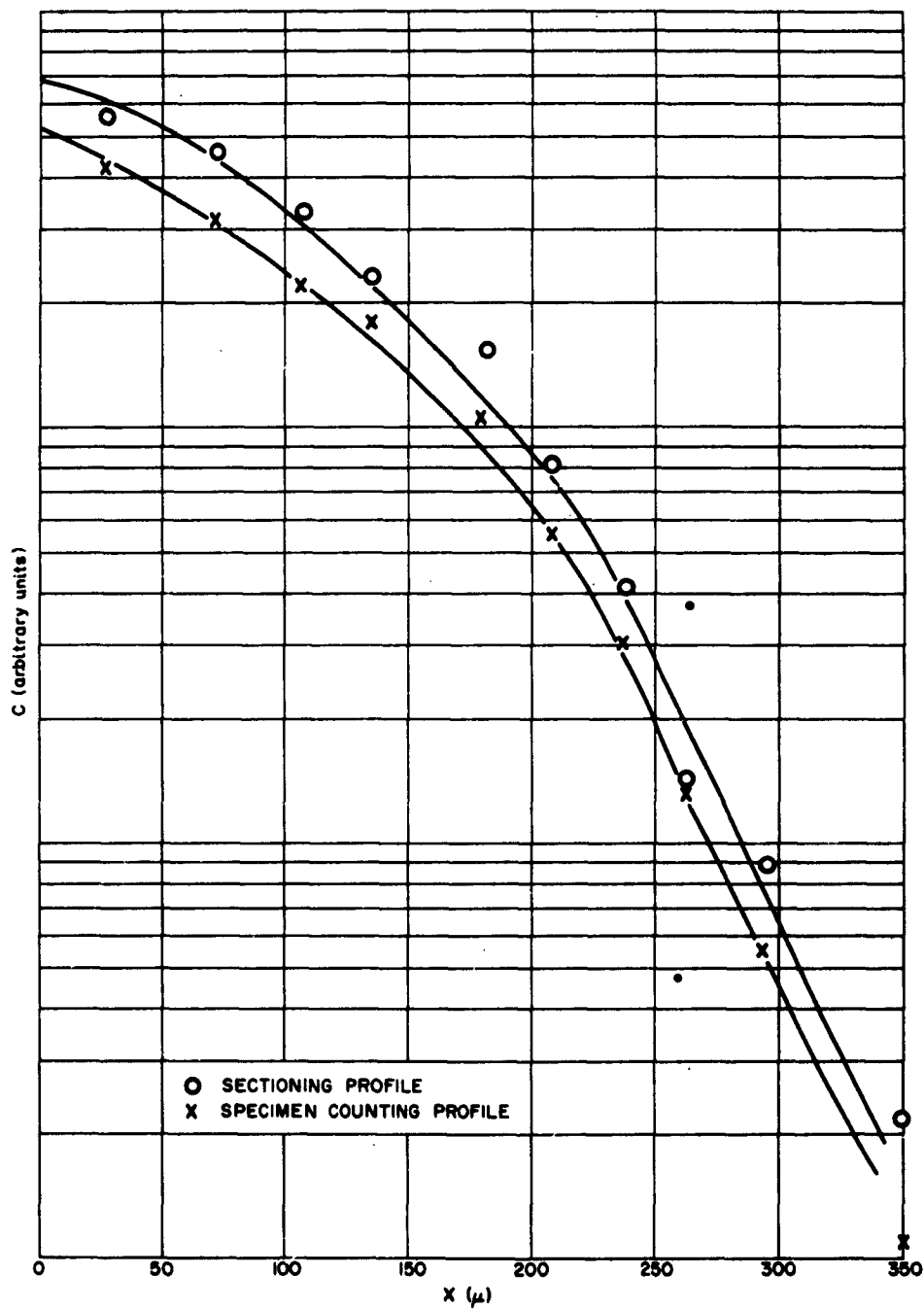


FIG. 1. CONCENTRATION PROFILES FOR DIFFUSION AT 804°C, $\ln C$ VERSUS X

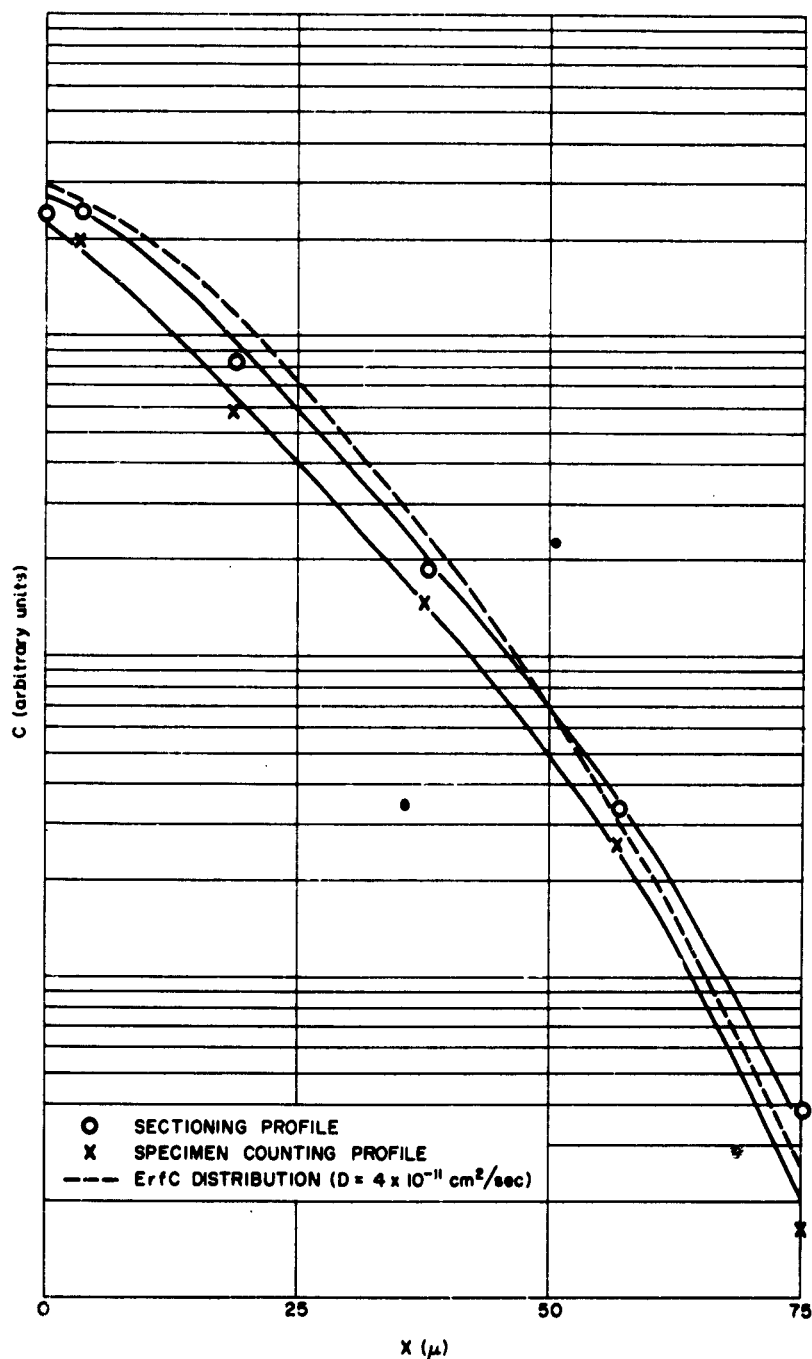


FIG. 2. CONCENTRATION PROFILES FOR DIFFUSION AT 607°C, $\ln C$ VERSUS X

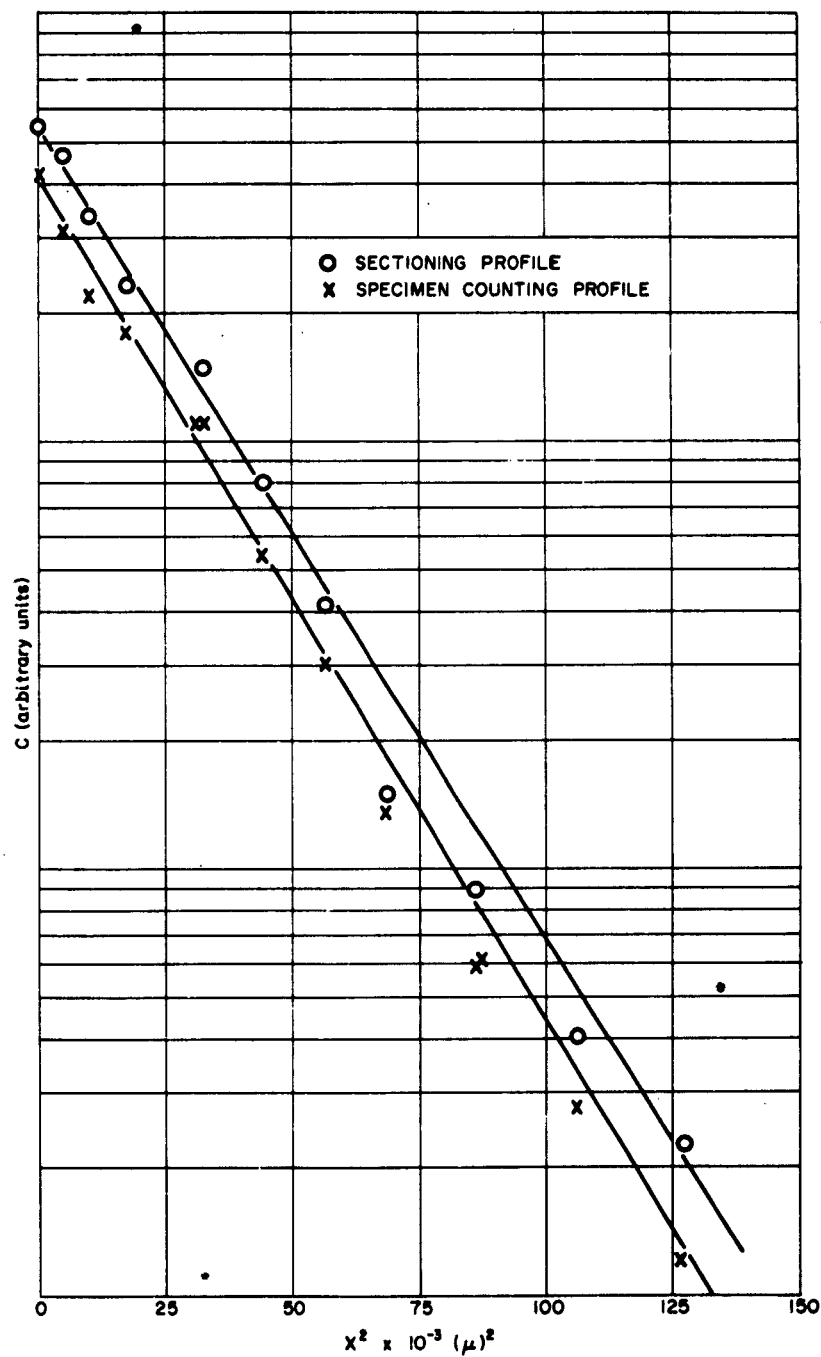


FIG. 3. CONCENTRATION PROFILES REPLOTED AS $\ln C$ VERSUS x^2

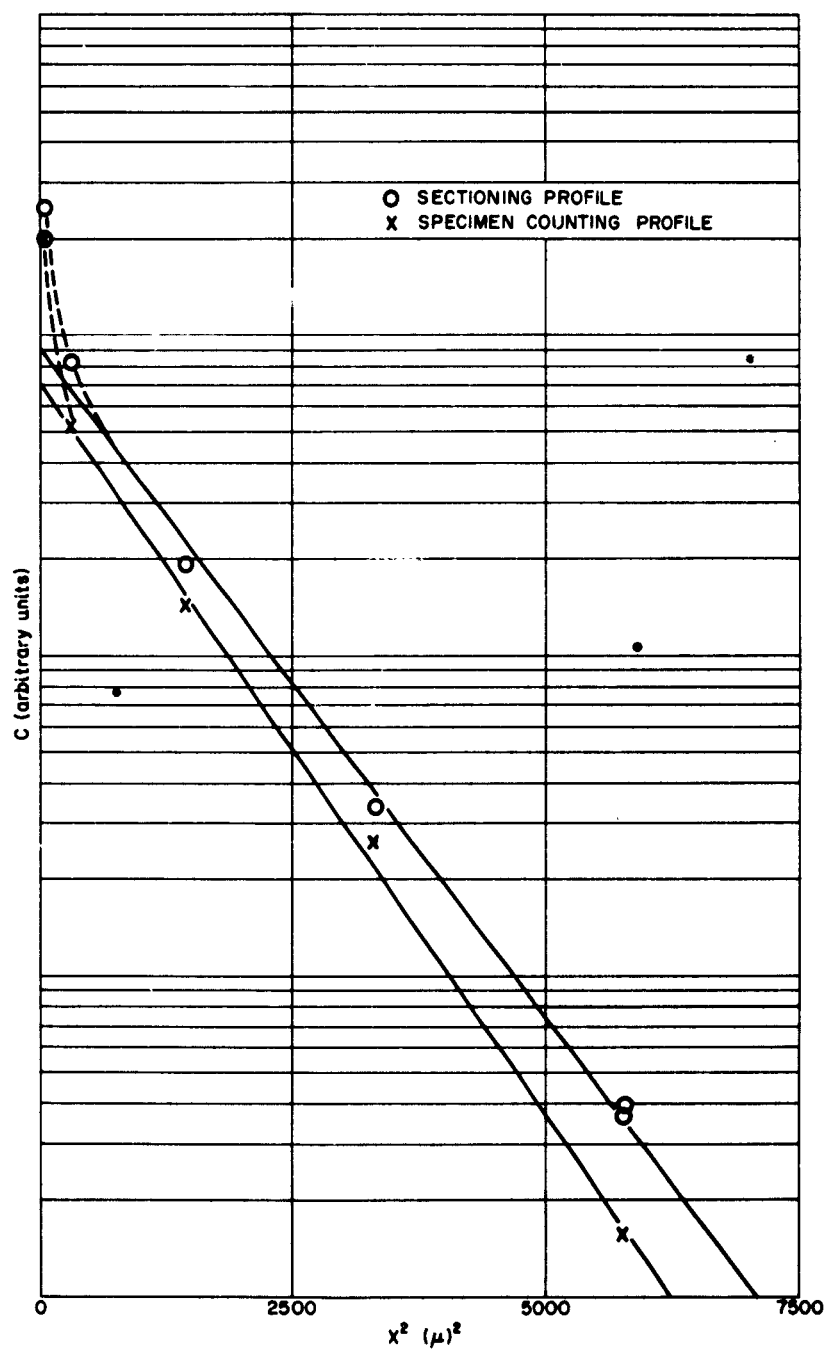


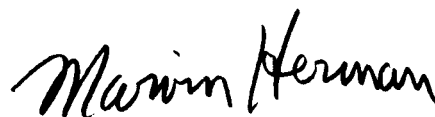
FIG. 4. CONCENTRATION PROFILES REPLOTED AS $\ln C$ VERSUS x^2

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close to a Gaussian distribution (Equation 3). The diffusion coefficient at 804°C is therefore determined as 3.5×10^{-9} cm²/sec from the slope of the curve in Figure (3).

On the other hand, the plot in Figure (4) is largely a straight line function, but deviates from linearity close to $X^2 = 0$. Although it is obvious that the curve in Figure 2 is not a Gaussian distribution, a diffusivity of 4.0×10^{-11} cm²/sec at 607°C may be estimated from the straight line portion of the curve in Figure 4. Now if one uses this D value and plots the erfc distribution (Equation 2) for this particular experimental condition as $\ln C$ vs. X , the dotted curve in Figure 2 is obtained. Since this curve is almost identical to the experimental curves, it may be concluded that the concentration profile of the experiment at 607°C is an erfc distribution.


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Approved by


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